



79132

NEW FORM OF CARBON

1 This is a continuation-in-part of copending United States Patent Application having Serial Number 575,254 that was filed on August 30, 1990.

5 BACKGROUND OF THE INVENTION

This invention relates to new forms of carbon as well as methods for the production and recovery thereof from carbon sources.

10 In 1985, Kroto et al. postulated the existence of a highly stable molecule composed of 60 carbon atoms based solely on mass spectroscopic analysis of vaporized graphite (H.W. Kroto, et al., Nature, Vol. 318, 162, 14 November 1985). More specifically, all that was observed  
15 was a peak in the mass spectra of said carbon vapor. However, Kroto et al. did not isolate any of said compound.

A model for this compound was proposed in which 60 carbon atoms are placed at the vertices of a truncated  
20 icosahedron forming a perfect "soccerball" structure. Subsequent thereto, many publications have strengthened the evidence for the existence of this molecule. The 60 carbon atom compound (hereinafter  $C_{60}$ ) was presumably  
25 produced in situ for the spectroscopic determination reported in these publications. Yet, to date, no one has been successful in verifying the existence of this molecule since no one has been successful in isolating the molecule in measurable amounts. Thus, no processes for producing recoverable amounts of this new compound  
30 have been described to the present time.

In the aforesaid publication by Kroto, et al., the authors proposed many uses for the new substance,  $C_{60}$  if it could be produced in quantity such as  $C_{60}$  transition metal compounds, e.g.,  $C_{60}Fe$ ; or halogenated  
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species like  $C_{60}F_{60}$  which might be a super lubricant;  
1 molecules including oxygen and lanthanum in the  $C_{60}$   
interior;  $C_{60}$  would provide a topologically novel  
aromatic nucleus for new branches of organic and  
inorganic chemistry; and  $C_{60}$  being especially stable and  
5 symmetrical provides possible catalyst and/or  
intermediate in modelling prebiotic chemistry.

Another form of carbon containing 70 carbon atoms  
( $C_{70}$ ) has also been postulated (Kroto, Chemistry in  
Britain, 40-45 (1990), Kroto, Science, 1139-1145 (1988)).  
10 Like the ( $C_{60}$ ) to date, no one has been successful in  
verifying the existence of the  $C_{70}$ . Heretofore, no one  
has been successful in obtaining the molecule in any  
appreciable amounts.

## 15 SUMMARY OF THE INVENTION

A process has now been developed for the  
production of recoverable amounts of  $C_{60}$  and  $C_{70}$ . The  
present new process is accomplished by evaporating carbon  
20 rods in an atmosphere of an inert quenching gas  
maintained at reduced pressure in a reactor therefor.  
This process produces a sooty carbon product which is  
graphitic carbon including a few percent of  $C_{60}$  and low  
levels of  $C_{70}$  which are recoverable from the product.  
25 However, an increase in the fraction of  $C_{70}$  molecules can  
be produced if the pressure is raised to greater than  
atmospheric pressures.

The recovery process is preferably accomplished  
by selective extraction of  $C_{60}$  and  $C_{70}$  with non-polar  
30 organic solvents from the sooty graphitic carbon.

The processes of the present invention produces  
 $C_{60}$  and  $C_{70}$  in recoverable amounts and permits  
realization of the proposed uses described hereinbefore.

**BRIEF** DESCRIPTION OF THE FIGURES

1 1

Fig. 1: ~~Micrograph~~ <sup>micrograph</sup> of typical crystals of the 98% C<sub>60</sub>, 2% C<sub>70</sub> material showing thin platelets, rods and stars of hexagonal symmetry.

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Fig. 2: X-ray diffraction of a microcrystalline powder of the 98% C<sub>60</sub>, 2% C<sub>70</sub> solid material. Inset at upper left is a single crystal electron diffraction pattern indexed with Miller indices compatible with the X-ray pattern, taken on a thin platelet as in Figure 1 with the electron beam perpendicular to the flat face.

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Fig. 3: Infrared absorption spectrum of an approximately 2 micrometer thick coating of the 98% C<sub>60</sub>, 2% C<sub>70</sub> material on a silicon substrate, referenced to a clean silicon substrate. Absorption is given as optical density = log<sub>10</sub> 1/T, where T is transmission. Apparent negative absorptions are due to the coating acting in part as a non-reflecting layer.

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Fig. 4: Visible-ultraviolet absorption spectrum of an approximately 0.1 micrometer thick coating of the 98% C<sub>60</sub>, 2% C<sub>70</sub> material on quartz. Shown at the bottom are positions and relative oscillator strengths for allowed transitions calculated for the C<sub>60</sub> molecule by Larsson, et al.

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DESCRIPTION OF THE PREFERRED EMBODIMENTS

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The first step in the production of C<sub>60</sub> and C<sub>70</sub> molecules is vaporizing carbon from any source containing carbon in its various forms, e.g., graphite, amorphous and glassy carbon. It is preferred that this vaporization takes place in an evacuated reactor (e.g., a bell jar). The carbon is vaporized by heating in the presence of an inert quenching gas. The carbon vapor is nucleated in the presence of the inert quenching gas to form smoke particles.

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In the production of  $C_{60}$  and  $C_{70}$ , any procedure  
1 for vaporizing carbon can be used, although the preferred  
method relies on the use of a high intensity electrical  
current with graphite rods as electrodes. These rods are  
constructed to permit vaporization of carbon at the tip  
5 of the rod to produce a high density vapor of carbon.  
For best results, the end of one of the rods is reduced  
in diameter so that the vaporization occurs at the  
reduced tip. The rods can be prepared using any of the  
various forms of carbon, such as graphite, amorphous and  
10 glassy carbon.

The inert quenching gas can be any of the usual  
inert gases such as the noble gas. Argon and helium are  
preferred, the latter being most preferred. Other inert  
gases commonly employed to provide a non-reactive  
15 atmosphere can also be used as quenching gas.

The amount of  $C_{60}$  and  $C_{70}$  produced from this  
carbon source is dependent upon the pressure of the  
quenching gas. At lower pressures relatively pure  $C_{60}$   
molecules can be produced in high yield with minor  
20 concentrations of  $C_{70}$ . For the production of  
predominantly  $C_{60}$  molecules, the pressure at which the  
quenching gas is maintained should be subatmospheric and  
preferably about 50-400 torr. Especially preferred is a  
pressure of approximately 100 torr. The use of any lower  
25 pressure may result in reduced yield of  $C_{60}$ .

However, as the pressure is raised, the ratio  
of  $C_{70}:C_{60}$  is also increased.

If the pressure is increased to at least two  
atmospheres, the greatest percentage of  $C_{70}$  product is  
30 formed. Theoretically, the pressure can be raised to any  
level just below the point where the reactor would

1 explode from the increased pressure. However, at the  
higher pressures, the yield of the overall product ( $C_{60}$   
and  $C_{70}$ ) is reduced even though the ratio of  $C_{70}:C_{60}$  is  
also increased. Therefore, as a practical consideration,  
5 the pressure of the quenching gas should not be greater  
than 10 atmospheres. The preferred pressure for  
maximizing the amount of  $C_{70}$  produced is 2-3 atmospheres.

The produced quenched vapor of carbon, i.e.,  
the smoked particles coats the internal surface of the  
10 reactor and of collecting substrates as black soot.  
These collecting surfaces are inert to the vaporized  
carbon. They can be transparent and/or coated with an  
inert metal. Examples include glass, or gold coated  
glass surfaces and the like. These collecting surfaces  
15 are located in the reactor in the path of the carbon  
smoke. The black coating can be removed by any suitable  
means, e.g., by scraping the solids from the coated  
surfaces. The  $C_{60}$  and  $C_{70}$  molecules can be removed from  
this collected quenched product by contacting said  
20 quenched product with an extracting solvent. In other  
words, the black soot is placed in a container containing  
the extracting solvent, or the extracting solvent is  
poured onto the black soot placed in a container. In  
either case, the  $C_{60}$  and  $C_{70}$  molecules become dissolved in  
25 the solvent, while the remainder of the black soot  
remains insoluble. The insoluble material is separated  
from the solution containing the  $C_{60}$  and  $C_{70}$  molecules,  
e.g., by decanting, or by filtration, and the like.

Suitable solvents include non-polar organic  
30 solvents, such as the alkanes containing 5-10 carbon  
atoms (e.g. pentanes, hexanes, heptanes, octanes),  
benzene and alkyl-benzenes (e.g. toluene, xylene), carbon  
disulfide, carbon tetrachloride, naphtha, 1,1,1-

1 trichloroethane, and the like. Simple solubility  
determinations using classical laboratory methods will  
permit selection of other suitable solvents. The  
preferred solvents are carbon disulfide, benzene, carbon  
5 tetrachloride and toluene. Especially preferred are  
benzene, carbon tetrachloride and carbon disulfide.

The product obtained contains a mixture of  $C_{60}$   
and  $C_{70}$ . As described hereinabove, the amounts of  $C_{60}$   
and  $C_{70}$  present is dependent upon the pressure used. If  
10 subatmospheric pressures are used, such as 50-400 torr,  
the product is predominately pure  $C_{60}$  with a minor amount  
of  $C_{70}$  present. Thus, when the collected product is  
dispersed in the extracting solvent, the product obtained  
is a mixture of  $C_{60}$  and  $C_{70}$ . For example, when the  
15 pressure is 100 torr, the product formed is about 98%  $C_{60}$   
and about 2%  $C_{70}$ . This product can be separated from the  
organic solvent solution by standard methods as by  
evaporation of the solvent or by dilution of the solvent  
solution with a non-solvent for  $C_{60}$ . The product can be  
20 crystallized by careful evaporation of the organic  
solvent or by sublimation procedures.

In a preferred embodiment of producing  $C_{60}$  and  
 $C_{70}$ , pure graphite rods are vaporized by passing high  
25 electrical current (either dc or ac) through narrowed  
tips of graphite rods. Electron beam, laser and RF  
heating can be used in lieu of electrical heating. This  
is done in a reactor (such as a bell jar) that has been  
evacuated, purged and filled with inert gas at or  
preferably below atmospheric pressure, e.g., pressures  
30 ranging from about 50 to about 400 torr. and even higher.  
The graphite rods are typically 1/4 inch in diameter with



1 about 1 cm length of one rod reduced in diameter to about  
5 mm. The electrical heating vaporizes the constricted  
tip of the graphite rod producing a high density vapor of  
5 carbon, which quickly condenses into a smoke consisting  
of very fine particles (of the order of 0.1 microns) of  
graphitic carbon with an admixture of a few percent of  
the desired  $C_{60}$  molecule. At this point in the process  
there is a heavy black coating on collecting substrates  
and/or on the walls of the chamber which can be easily  
10 scraped off for the recovery step.

For recovery, the sooty product is treated with  
benzene to provide a brownish-red solution. After  
separation of the undissolved graphitic carbon, the  
benzene solution is evaporated to obtain microcrystalline  
15 product. Alternatively, the product can be sublimed from  
the sooty carbon at  $300^{\circ}$  to  $400^{\circ}\text{C}$ . and the sublimation  
product obtained by condensation on a conventional  
substrate.

When the pressure of inert quenching gas is 100  
20 torr, the product formed is 98%  $C_{60}$  and 2%  $C_{70}$ . This  
product, as obtained from the solvent extract of the  
sooty graphitic carbon, is a dark brown to black  
crystalline material. When obtained by sublimation in  
vacuum or inert atmosphere, the product is obtained as a  
25 brown to gray coating depending on thickness.

On analysis by mass spectroscopy, the spectrum  
clearly shows a strong peak at mass 720 amu (i.e., the  
mass of  $C_{60}$ ) and a clean peak at 840 amu (i.e., the mass  
of  $C_{70}$ ). Significant differences in the spectra occur  
30 only in the abundances in the mass domain lower than 300  
amu. Most of these differences seem to originate from  
the different ionization techniques in the mass  
spectrometer and from the different kinds of sample  
desorption. So far, the cleanest mass spectra have been

1 obtained when the material was evaporated and ionized in  
the vapor phase by electrons. In such spectra the mass  
range above 40 amu is dominated by the  $C_{60}$  mass along  
with its expected isotope lines. The only other large  
5 mass found in any abundance corresponds to  $C_{70}$ , with a  
ratio of  $C_{70}$  to  $C_{60}$  of about .02.

Studies by optical microscopy of the  $C_{60}$   
material which is left after evaporating the benzene  
solution show a variety of what appear to be crystals --  
10 mainly rods, platelets, and star-like flakes. Figure 1  
shows a micro-photograph of such a crystal assemblage.  
All crystals tend to exhibit six-fold symmetry. In  
transmitted light they appear red to brown in color; in  
reflected light the larger crystals have a metallic  
15 appearance, whereas the platelets show interference  
colors consistent with an index of refraction of about 2.

The platelets can be rather thin and thus are ideally  
suited for electron diffraction studies in an electron  
20 microscope. (See the insert in Figure 2).

In order to determine if the  $C_{60}$  molecules form  
a regular lattice electron, x-ray diffraction studies on  
the individual crystals and on the powder were carried  
out. A typical X-ray diffraction pattern of the purified  
25  $C_{60}$  powder is shown in Figure 2. To aid in comparing the  
electron diffraction results with the X-ray results the  
electron diffraction pattern is inserted into the corner  
of Figure 2. From the hexagonal array of diffraction  
spots indexed as shown in the Figure, a d-spacing of 8.7  
30 Å was deduced corresponding to the (100) reciprocal  
lattice vector of a hexagonal lattice. The most obvious  
correspondence between the two types of diffraction is  
between the 5.01 Å peak of the X-ray pattern and the



1 (100) spot of the electron diffraction pattern, which  
 gives a spacing of about 5.0 Å. Assuming that the C<sub>60</sub>  
 molecules are behaving approximately as spheres stacked  
 in a hexagonal close packed lattice with a c/a ratio of  
 5 1.633, d-spacings can be calculated. The results are  
 shown in Table I.

Table I: X-Ray Diffraction Results and  
 Assignments For a Hexagonal Lattice Using  
 a = 10.02 Å, c = 16.36 Å

$$\frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{2}{c^2}$$

15	Measured 2θ (degrees)	Measured d-spacing (Å)	Calculated d-spacing (Å)	Assignment (hkl)
	10.2 shoulder	8.7	8.68	(100)
20	10.81	8.18	8.18	(002)
			7.67	(101)
	17.69	5.01	5.01	(110)
	20.73	4.28	4.27	(112)
	21.63	4.11	4.09	(004)
25	28.1	3.18	3.17	(114)
	30.8	2.90	2.89	(300)
	32.7	2.74	2.73	(006)

30 The values derived from this interpretation are  
 a = 20.02 Å and c = 16.36 Å. The nearest neighbor  
 distance is thus 10.02 Å. For such a crystal structure  
 the density is calculated to be 1.678 g/cm<sup>3</sup>, which is  
 consistent with a value of 1.65 +/- .05 determined by  
 suspending crystal samples in aqueous GaCl<sub>3</sub> solutions of

1 known densities. Although the agreement shown in Table 1  
is good, the absence of the characteristically strong  
(101) diffraction in hcp and the broad continuum in  
certain regions suggest a less than perfect crystalline  
5 order. Furthermore, X-ray diffraction patterns obtained  
on carefully grown crystals up to 500 micrometers in size  
with well developed faces yielded no clear spot pattern  
(in contrast to the electron diffraction pattern on  
micron-size crystals). It thus appears that these larger  
10 crystals do not exhibit long range periodic order in all  
directions.

A likely explanation for the unusual  
diffraction lies in the disordered stacking arrangement  
of the molecules in planes normal to the c-axis. It is  
15 well known that the position taken by spheres in the  
third layer of stacking determines which of the  
close-packed structures occurs, the stacking arrangement  
in fcc being ABCABC while that in hcp is ABABAB. If the  
stacking sequence varies, the X-ray lines due to certain  
20 planes will be broadened by the disorder while other  
lines will remain sharp. Such disordered crystalline  
behavior was observed long ago in the close packed  
structure of cobalt, where X-ray diffraction lines such  
as (101), (102) and (202) were found to be substantially  
25 broadened due to the stacking disorder. Reflections from  
planes such as (002) remain sharp since these planes have  
identical spacings in both fcc and hcp structures. A  
general expression for which peaks are broadened by this  
kind of disorder have been given in terms of Miller  
30 indices (h,k,l) as  $h - k = 3t \pm 1$ ,  $l \neq 0$ , where t is an  
integer. None of these broadened reflections are  
apparent in the X-ray pattern of Figure 2. This may  
explain the weakness of the characteristically strong

1 (101) peak. Whether or not this stacking disorder is  
related to the presence of the possibly elongated  $C_{70}$   
molecules is yet to be determined.

5 In small crystals at least, the  $C_{60}$  molecules  
appear to be assembling themselves into a somewhat  
ordered array as though they are effectively spherical,  
which is entirely consistent with the soccer ball  
hypothesis for their structure. The additional diameter  
10 over the calculated 7.1 Å value for the carbon cage  
itself must represent the effective van der Waals  
diameter set by the repulsion of the pi electron clouds  
extending outward from each carbon atom. Scanning  
tunnelling spectroscopy of the  $C_{60}$  molecules clearly  
shows the spherical nature of the  $C_{60}$  molecules.

15 Some scanning tunnelling microscope images of a  
carbon sample prepared in accordance with the procedure  
described hereinabove at pressures of helium at 100 torr  
show a spherical molecule of twice the diameter of the  
 $C_{60}$  molecules. This is evidence of the existence of a  
20 caged molecule containing 240 carbon atoms or a  $C_{240}$   
molecule.

Samples were prepared for spectroscopy by  
subliming pure material onto transparent substrates for  
transmission measurements. Depending on the pressure of  
25 helium in the sublimation chamber, the nature of the  
coatings can range from uniform films (at high vacuum) to  
coatings of  $C_{60}$  smoke (i.e., sub-micron microcrystalline  
particles of solid  $C_{60}$ ) with the particle size depending  
to some extent on the pressure.

30 Figure 3 shows the transmission spectrum of an  
approximately 2 micrometer thick  $C_{60}$  coating on a silicon  
substrate. The infrared bands show the four most intense  
lines at 1429, 1183, 577, and 528  $\text{cm}^{-1}$ , with no

1 underlying continuum remaining from the soot. In early  
tries at purifying  $C_{60}$  material, the infrared spectrum  
showed a strong band in the vicinity of 3.0 micrometers,  
which is characteristic of a CH stretching mode. After  
5 much effort, this contaminant was successfully removed by  
washing the soot with ether and using distilled benzene  
in the extraction. The spectrum in Figure 3 was obtained  
when the material cleaned in such a manner was sublimed  
under vacuum onto the substrate. The spectrum shows very  
10 little indication of CH impurities.

The presence of only four strong bands is what  
is expected for the free, truncated icosahedral molecule  
with its unusually high symmetry. Also present are a  
number of other weak infrared lines which may be due to  
15 other causes, among which may be absorption by the  $C_{70}$   
molecule or symmetry breaking produced, for example, by  
isotopes other than  $C^{12}$  in the  $C_{60}$  molecule or by mutual  
interaction of the  $C_{60}$  molecules in the solid.  
Noteworthy, are weaker features at about 2330 and 2190  
20  $cm^{-1}$  which are located in the near vicinity of the free  
 $CO_2$  and CO stretching modes. This may imply some  
attachment of  $CO_2$  or CO to a small fraction of the total  
number of  $C_{60}$  molecules. Another noteworthy effect can  
be observed in the feature at  $675\ cm^{-1}$ , which is weak in  
25 the thin film samples but almost as strong as the four  
main features in the crystals. This vibrational mode may  
be of solid state rather than molecular origin.

Figure 4 shows an absorption spectrum taken on  
a uniform film coated onto a quartz glass substrate. The  
30 ultraviolet features are no longer obscured by the  
graphitic carbon background as in our previous spectra.  
Broad peaks at 216, 264 and 339 nm dominate the spectra.  
Weaker structures show up in the visible, including a

1 plateau with ends at about 460 and 500 nm and a very weak  
peak near 625 nm. At the bottom of Figure 4 are shown  
positions and relative oscillator strengths taken from  
Larsson, et al. (Chem. Phys. Lett. 137, 501-504)  
5 calculated for the  $C_{60}$  molecule. This reference also  
shows a variety of forbidden bands with the lowest energy  
ones in the vicinity of 500 nm. There seems to be a  
rough correspondence between the present measurements on  
thin films and the allowed transitions predicted for the  
10 molecule. There was no band at 386 nm in our films of  
 $C_{60}$ , as disclosed by Heath, et al. (J. Chem. Phys. 87,  
4236-4238 (1987)) using a laser depletion spectroscopy  
method and attributed to the  $C_{60}$  molecule. Quite similar  
spectra to that in Figure 4 have been recorded for  
15 microcrystalline coatings deposited at helium pressures  
of 100 torr, for example. The peaks occur at the  
slightly shifted positions of 219, 268, and 345 nm.

The  $C_{70}$  molecule is larger than the  $C_{60}$   
molecule. The  $C_{70}$  molecule shows a molecular ion peak at  
20 840 amu. Furthermore, a noticeable peak in the  
ultraviolet spectrum of the  $C_{70}$  molecule taken on a  
uniform film coated onto a quartz glass substrate is  
exhibited at about 216 nm. This is a broad peak.  
Surprisingly, it appears that the  $C_{70}$  molecule is more  
25 stable than  $C_{60}$ .

Thus, using the procedures described  
hereinabove, at quenching pressures of less than 1  
atmospheric pressure and especially at pressures of  
50-400 torr, a product is produced which is predominantly  
30  $C_{60}$  and <sup>contains</sup> ~~contains~~ minor amounts of  $C_{70}$ . The  $C_{60}$  product  
can be used or can be further purified.

Further purification and separation of  $C_{60}$  and  
 $C_{70}$  can be made by many conventional techniques known to

1 one skilled in the art, e.g., fractional crystallization,  
column chromatography, capillary electrophoresis, HPLC,  
preparative thin-layer chromatography, and the like.

5 Because the molecular figuration of  $C_{60}$  and  $C_{70}$   
are different, the attractive intermolecular forces are  
different which allows for the two molecules to be  
readily separated.

10 Furthermore, the solubility of  $C_{60}$  and  $C_{70}$  in  
pure solvents and mixed solvents are also different from  
each other, which also makes the two compounds separable  
by using conventional techniques known to one skilled in  
the art, such as crystallization, extraction, and the  
like.

15 For example, pure  $C_{60}$  and pure  $C_{70}$  molecules  
can be isolated as follows. The black sooty mixture of  
 $C_{60}$  and  $C_{70}$  which is produced according to the procedure  
described hereinabove is placed in the extracting  
solvent, such as benzene. The insoluble residue is  
removed and the resulting benzene solution containing  $C_{60}$   
20 and  $C_{70}$  molecules is concentrated. The  $C_{60}$  and  $C_{70}$   
solution is added to a packed column with an adsorbent,  
such as alumina. The column is eluted with an eluent  
such as benzene or a mixture of benzene and toluene.  
Various fractions of set volume e.g., 10 mL, are  
25 collected. The eluent i.e., the solvent is removed from  
each fraction such as by evaporation to dryness. The  
fractions with product will contain microcrystals, the  
identity of which can be confirmed by spectroscopy, e.g.,  
mass spectroscopy.

30 Thus, the process of the present invention can  
produce a product which is predominantly  $C_{60}$ , which, if  
desired, can be further purified by the purification and  
separation techniques described hereinabove.



1 Furthermore, the present invention contemplates  
two different variations of the procedure described  
hereinabove to make  $C_{70}$  molecules. First, if  
subatmospheric pressures of quenching gases are used in  
5 the initial step, small amounts of  $C_{70}$  are produced,  
which can be separated from the  $C_{60}$  molecules using the  
purification techniques described hereinabove. However,  
if the pressure of the quenching gas is raised to at  
least 2 atmospheres, after separation and purification, a  
10 greater percentage of substantially pure  $C_{70}$  would be  
produced from each vaporization of carbon.

The present new products,  $C_{60}$ ,  $C_{70}$ , or mixtures  
thereof have the similar utilities as graphite. However,  
they are particularly valuable for forming products of a  
15 higher order of stability than those formed from  
graphitic carbon, and can be processed into formed or  
molded products such as  $C_{60}$  fibers,  $C_{70}$  fibers, or  
mixtures thereof using standard processing techniques.  
In this regard, free-flowing, particulate  $C_{60}$  and  $C_{70}$  are  
20 of special value particularly for use in producing molded  
products, especially those extended in at least one  
direction.  $C_{60}$ , and  $C_{70}$  are also useful for producing a  
low temperature  $C_{60}$  vapor ( $300^{\circ}$ - $400^{\circ}$ C.) and  $C_{70}$  vapor of  
the respective molecules to produce low temperature  
25 atomic and molecular beams of carbon which is not now  
possible using graphite as the carbon source. Further,  
the synthesis of compounds such as  $C_{60}H_{60}$  and  $C_{60}F_{60}$  can  
be accomplished by introducing hydrogen and fluorine,  
respectively, into a reactor containing  $C_{60}$  vapor.  
30 Furthermore, the  $C_{60}$  product and the  $C_{70}$  product may be  
used as an industrial paint pigment or as a lubricant.  
Moreover, since the  $C_{60}$  and  $C_{70}$  molecule are hollow, they  
could be used for binding and/or storing molecules e.g.,  
toxic material.

EXAMPLE 1

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$C_{60}$ -containing carbon dust was produced in a conventional bell-jar carbon evaporator which was first evacuated to  $10^{-4}$  torr by either an oil diffusion pump or a turbo pump, both equipped with liquid nitrogen traps, and then filled with an inert quenching gas. Helium and argon were used at pressures ranging up to 400 torr. Then graphite rods (as previously described herein) were evaporated using a current of about 100 amps (either AC or DC).

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The smoke which formed in the vicinity of the evaporating carbon rods was collected on substrates which were placed within 5 cm to 10 cm of the evaporating carbon rods.

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The evaporator was opened after a cool down period of 10-30 min. and the carbon dust samples removed by scraping substrate surfaces and the internal surfaces of the bell-jar. After washing with ether, the collected dust samples were then extracted with benzene to produce a wine-red to brown solution. On evaporation of the solution,  $C_{60}$  was obtained as a microcrystalline residue.

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The crystals were sublimed by heating in vacuo or in a quenching inert gas to 400°C. and collected on a substrate. The sublimed product was brown to gray in color.

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In powder form, the present new carbon allotrope is brownish-red.

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EXAMPLE 2

1           The procedure of Example 1 is repeated except,  
in the original step, the graphite rods are evaporated at  
2 or more atmospheres of helium pressure in the chamber.  
The product obtained from this procedure contains a  
5 greater percentage of  $C_{70}$  than does the product in  
Example 1.

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EXAMPLE 3

1 Pure C<sub>60</sub> and pure C<sub>70</sub> are obtained as follows:

5 The C<sub>60</sub> and C<sub>70</sub> mixtures prepared in either Examples  
1 or 2 are dissolved in benzene and added to an alumina  
column. Using benzene as the eluent, the fractions are  
collected and each elute fraction is evaporated to  
dryness. The presence of C<sub>60</sub> or C<sub>70</sub> in the fraction can  
be determined by taking mass spectroscopy thereof.

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1       The above embodiments and examples are given to  
illustrate the scope and spirit of the instant invention.  
These embodiments and examples are within the  
contemplation of the present invention. Therefore, the  
present invention should be limited only by the appended  
5       claims.

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